

# Influence of pH Value of the Oral Cavity on Biaxial Flexural Strength of Layered Ceramics

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## Abstract

In this paper, effect of pH value of oral cavity on the bi-axial flexural strength of veneering porcelain containing zirconia is investigated by means of cyclic loading tests. In the experiments, standardized disc specimens ( $n=240$ , 16 mm  $\times$  1.6 mm) are pre-loaded with 600 N for 10,000 cycles and stored in a dry or wet (pH: 4, 7 and 8) environment prior to biaxial flexural strength testing (ISO 6872 standard). Hsueh's simple solutions are used to calculate stress distribution along the direction of thickness of bilayered discs subjected to biaxial moment loading. The fracture mode and location of cracks are identified by optical microscopy. A Weibull statistical approach is used to evaluate the reliability of the failure strength data. It is found that the flexural strength of specimens tested under dry conditions is approximately 20 ~ 50 % stronger than that of specimens soaked in the solution. The flexural strength of specimens soaked in an acid solution is approximately 30 % greater than that of specimens soaked in an alkaline solution. The Weibull modulus of specimens is 17.73, 17.88, 10.53 and 17.92 in pH 4, pH 7, pH 8 and dry environment respectively. Thus, the structural reliability of specimens used in this work are better when they were soaked in pH 4, pH 7 and was in dry environment than that in pH 8. The test results of bi-axial flexural strength in different storage media may provide a guide to the clinical application of all-ceramics dental crowns.

## Keywords

*Bi-axial Flexural Strength; Cyclic Loading; All-ceramics Dental Crown*

## Introduction

All-ceramic materials are increasingly becoming a preferred alternative to metal-ceramic restoration due to their excellent esthetics, chemical stability, and biocompatibility. The brittleness and low tensile

strength of conventional glass-ceramics, however, restrains its long-term clinical application in restorations. Compared to other dental glass ceramic systems, zirconia-based ceramics is used clinically for restorations with up to four units. The problem is that current technologies can neither make zirconia frameworks as translucent as natural teeth nor provide internal shade characterization or facilitate customized shading (Luthy et al, 2005; Zarone et al, 2011). As a result, zirconia cores (or frameworks) must be veneered with porcelain to achieve acceptable esthetics (White et al, 2005). In clinical applications, the veneering porcelain has already proved to be the weakest link in such reconstructions (Fischer et al, 2008; Raigrodski et al, 2006; Sailer et al, 2007a;b). Chipping of the veneer during mastication under wet conditions is identified as the main reason for failure, at the rate of 15.2 % after a service time of  $35.1 \pm 13.8$  months (Sailer et al, 2007a).

On one hand, all-ceramic restorations have less longevity than porcelain-fused-to-metal (PFM) fixed partial dentures (FPDs), as the bilayered ceramic materials have low flexural strength. On the other hand, the survival rates of all CAD/CAM ceramic single tooth restoration are equal to PFM-restorations while FPDs show higher complications rates (Land and Hopp, 2010). So, the study of mechanical properties of all-ceramic restorations is necessary.

In recent years many testing and modeling methodologies have been developed to characterize mechanical properties of dental restorative materials (Hsueh, 2006; Qin and Swain, 2004; Sailer et al, 2007b; Thompson, 2000; Zeng et al, 1998). Those tests did not contradict each other, but rather reflect the mechanical

properties of the dental restorative materials under different conditions (Chai et al, 2007). Testing specimens for bi-axial flexure were reported to be easier to prepare reproducibly, to match more closely the size of the clinical restoration, and to be less sensitive to edge defects than those for uniaxial flexure (Ban and Anusavice, 1990; Pagnano et al, 2005; Rosenstiel et al, 1993). All-ceramic dental crowns are usually fabricated into an esthetically pleasing multilayered structure. Because the flexural strength formulas of ISO 6872 (International Organization for standardization, 2008) and ASTM 1499 do not adapt to multilayered specimens (Zeng et al, 1998), Hsueh's simple solution (Hsueh and Kelly, 2009) is employed in this study.

As stated by Zeng et al. (1996), mean flexural strength values are related not only to the test method but also to the test environment (dry, wet, and repetitive stress). To the authors' knowledge, there are no available reports on the flexural strength of veneering ceramics for zirconia by way of cyclic loading test under different pH conditions. This is the motivation of the study whose objective is to evaluate the effect of humidity and pH value on the biaxial flexural strength of bilayered ceramics experiencing cyclic loading. The results may serve as a guide to clinical applications of all-ceramics dental crowns and to developing new-style all-ceramics dental crowns. The zirconia ( $t\text{-ZrO}_2$ ) used in this study is obtained from Guang Xi Cercon Corporation and the content of  $\text{ZrO}_2$  is 95%. A Weibull statistical methodology is utilized to identify both the difference in magnitude of the calculated failure stress and the distribution of the failure stress data.

## Materials and Methods

The materials used in this work were obtained from Guang Xi Cercon Corporation and their properties were provided by the manufacturer (see Table 1).

As indicated in (Pinto et al, 2008), the normal pH of saliva varies from 6.8 to 7.2. However, when an individual takes foods or drinks into the mouth, those foods may produce acidic or alkaline environments, resulting in acidic or alkaline environment. As a response to this phenomenon, the saliva flux may increase in order to provide bicarbonate ions that will adjust the oral pH to the normal level again (within buffering capacity). But, when the buffering capacity is exceeded, the oral pH cannot be adjusted to the normal level by buffering capacity of saliva flux. Thus, the oral pH may vary even from 4 to 8. To assess the

effect of extreme pH value of the oral cavity on the biaxial flexural strength of bilayered ceramic, this study considers three experimental groups (pH=4, pH=7, and pH=8) and one control group (dry) with 60 specimens each. The specimens in each group are subjected to 10,000 cyclic loadings.

TABLE 1 MATERIAL PROPERTY FROM THE MANUFACTURER'S DATA

Material	Cercon base	Ceron Ceram S powder dentine	Ceron Ceram S powder liner
Manufacturer	Degudent GmbH, Hanau-Wolfgang, Germany		
Batch	18002346	60668, 63481	
Composition	$\text{ZrO}_2$ ( $\text{HfO}_2$ ) : 95 (<2 $\text{HfO}_2$ ); $\text{Y}_2\text{O}_3$ : 5; $\text{Al}_2\text{O}_3$ + other oxides < 1 (+ $\text{SiO}_2$ )	$\text{SiO}_2$ : 60.0 ~ 70.0; $\text{Al}_2\text{O}_3$ : 7.5 ~ 12.5; $\text{K}_2\text{O}$ : 7.5 ~ 12.5; $\text{Na}_2\text{O}$ : 7.5 ~ 12.5	$\text{SiO}_2$ : 60.0 ~ 70.0; $\text{Al}_2\text{O}_3$ : 7.5 ~ 12.5; $\text{K}_2\text{O}$ : 7.5 ~ 12.5; $\text{Na}_2\text{O}$ : 7.5 ~ 12.5
Modulus / GPa	210	60~70	60~70
Flexural strength / MPa	900	80~90	80~90
CTE / $10^{-6}\text{K}^{-1}$	10.5	9.5	9.5

Note: Thermal expansion coefficient is in  $10^{-6}\text{K}^{-1}$  and valid between 25 and 500°C

## Preparation of Zirconia Core

In the preparation of specimens, discs (16 mm diameter and 0.8 mm thickness) are cut from cercon zirconia blocks by an electrical high-precision saw (Labcut1010, EXTEC, USA), and then polished by grinding and polishing machine (Labopol-6, Struers, Denmark), 320 and 600 grit silicon carbide paper prior to sintering. This is followed by a process of sintering to full density according to the manufacturer's instructions (1350 °C for 6 h) by Cercon heat (DeguDent GmbH, Germany). It should be mentioned that the specimens are cut oversized because approximately 20 % shrinkage will occur during the process of sintering. Disc dimensions are measured with a high-precision micrometer caliper with an accuracy of  $\pm 0.01$  mm. The total time of the whole process of zirconia core preparation, including heating, sustaining and cooling, is approximately 8 hours. Nominally identical disc shaped specimens containing Y-TZP core and with the dimension of 16 mm diameter and 0.8 mm thickness are then produced.

Finally, the specimens of 0.8 mm thickness are treated like fixed partial dentures.

### **Preparation of Core Veneer Specimen**

Regarding the core veneer specimens, the core is sandblasted with 110  $\mu\text{m}$   $\text{Al}_2\text{O}_3$  particles for 30 s at 2.5 bar pressure (Rocatec-Pre, Espe, Seefeld, Germany) after sintering. Before veneering, all core specimens are cleaned with ultrasonic cleaner and air-dried. According to the procedure of manufacturers (Cercon, DeguDent GmbH, Germany), the veneering porcelain is built up to the final dimensions (1.6 mm  $\times$  16 mm) in a metal mold by Programat furnaces (P200, Ivoclar Vivadent Inc., Liechtenstein).

### **Specimen Soaking**

The solutions used in specimen soaking consist of following ingredients: 0.78 g  $\text{Na}_2\text{HPO}_4 \bullet 2\text{H}_2\text{O}$ ; 0.4 g KCl; 0.4 g NaCl; 0.795 g  $\text{CaCl}_2 \bullet 2\text{H}_2\text{O}$ ; 0.005 g  $\text{Na}_2\text{S} \bullet 2\text{H}_2\text{O}$  and 1 g Urea. Three solutions are produced with pH of 4, 7 and 8. The pH value of the solution is adjusted to 4 by adding HCl and to 8 by adding NaOH. The pH values of all storage media are measured using a pH meter (WPH2, DWYER, USA). The 180 specimens used in the test are equally divided into the three groups and each group is immersed in one solution for 2 weeks at 37 °C. And the other 60 specimens used as control group are not soaked in the solutions. In order to simulate laminated composite of all-ceramic crown, specimens are cemented to polyethylene fiber resin posts by epoxy resin after soaking. The polyethylene fiber resin posts, having an elastic modulus similar to that of dentin, are all incubated in water for more than 2 weeks before cementation to allow for hydroscopic expansion. After soaking, all of the specimens are used for fatigue test and flexural strength test in order to evaluate the influence of pH on fatigue damage and biaxial flexural strength of bilayered ceramics.

### **Fatigue Tests**

The cyclic loading applied on the top surface (veneering porcelain) of the bilayered discs is delivered with a spherical tungsten carbide indenter ( $r = 3.18$  mm) by a mechanical cycling machine (AG-20I, Shimadzu Corp, Japan) that can generate mechanical forces similar to those occurring in the chewing cycle. The specimens are tested with a controlled stroke profile: maximum load (biting force) of  $P_m = 600$  N for 10,000 times (Pittayachawan et al, 2009); loading and unloading rates being 1000 N/sec; and a chewing

frequency of 1/6 Hz. The steps of each load cycle consist of the indenter coming into contact with the specimen, loading to a maximum, holding for 0.2 sec, unloading, and lifting off (0.5 mm) from the specimen surface, then loading to a minimum load of 50 N. Fatigue tests are stopped after a pre-set number of loading cycles. It is estimated 65% specimens fractured during fatigue testing and the equivalent stress on zirconia was 900MPa during fatigue testing. We noticed that while the core (zirconia) can survive at 900MPa cycling load, the top surface material (porcelain veneer) might fail at a load level below 900MPa. It should be mentioned that the zirconia plays an important role as support materials in improving failure behaviour of brittle layer structures. The specimen is monitored for surface damage with an optical microscope (LV150, Nikon, Japan). All specimens are selected for evaluating the extent of subsurface damage.

### **Biaxial Flexure Strength Measurement**

After fatigue testing, flexural strength tests are carried out for those un-fractured specimens using the piston-on-three-balls method according to ISO standard 6872 (International Organization for standardization, 2008) in a universal testing machine (AG-20I, Shimadzu Corp, Japan). The biaxial strength tests conducted under the same environment condition (in air). The disk containing specimens is supported by three balls. The balls have a diameter of 4 mm each and are positioned at an angle of 120° to each other, forming a circum-circle 12 mm in diameter. The piston (diameter 1.5 mm) acts on the center of the specimen at a speed of 1 mm/min until the specimen is broken. A closed-form solution in the guidelines of Hsueh (2009) is used to calculate the biaxial flexural strength of different regions of specimens. To investigate the failure mode, the fracture fragments of each bilayered specimen are reassembled and inspected by an optical microscope (LV150, Nikon, Japan).

### **Statistical Analysis**

Biaxial flexural strength data corresponding to each group are analyzed using a one-way analysis of variance (ANOVA) and comparison is made by way of Tukey's post hoc test at a pre-set significance level of 5 %. In addition, in order to calculate the Weibull modulus, the biaxial flexural strength data of each group are listed in an ascend order. Then the Weibull modulus is calculated by the equation (Weibull, 1951):

$$P_f = 1 - \exp [-(\sigma/\sigma_0)^m]$$

where  $\sigma_0$  is a constant,  $m$  is the Weibull modulus,  $\sigma$  is the biaxial flexural strength,  $P_f$  is the fracture probability of the 60 specimens in each group. The variables  $P_f$  and  $\sigma$  are transformed to  $\ln [1/(1-P_f)]$  and  $\ln \sigma$ , respectively. This is the double natural logarithm of  $1/(1-P_f)$  and the natural logarithm of  $\sigma$ , which is usually used for Constructing plots with  $\ln [1/(1-P_f)]$  as the ordinate, a corresponding  $\ln \sigma$  as the abscissa, and the slope of the plot is equal to  $m$  (International Organization for standardization, 2008).

## Results

### Fatigue Damage

After fatigue test, the statistical analysis is performed using the testing results of subsurface damage. The extent of damage can be divided into several cases, which include conspicuous crack and inconspicuous crack. The results of fatigue tests confirm the influence of pH on fatigue damage of bilayered ceramics to be significant, especially in alkaline environment; where the conspicuous damage is visibly increasing by 50% in contrast to that in dry condition (see Fig. 1).

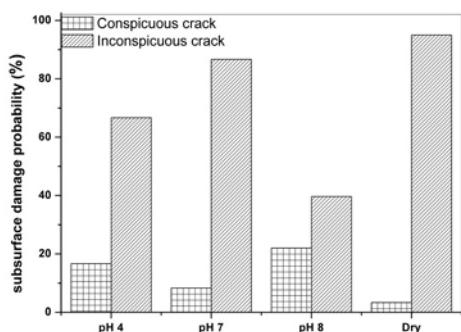


FIGURE 1 RESULTS FOR THE STATISTICAL ANALYSIS OF SUBSURFACE DAMAGE OF PORCELAIN / ZIRCONIA BILAYERED CERAMIC DISCS

### Biaxial Flexural Strength

Fig. 2 shows that discs have the highest value of flexural strength in the dry condition and the lowest value of flexural strength in pH 8. The flexural strength of the discs in the dry condition is approximately 7% higher than that in pH 7, 20% higher than that in pH 4 and 50% higher than that in pH 8. The strength values of the material at the bottom of the discs are 95% higher than those of the materials at the top of discs regardless of storage medium. While the flexural strength of specimens in acid solutions is approximately 30% stronger than those were soaked in alkaline solutions. The mean failure

stresses at the interface 1(the contact surface which is near to zirconia between zirconia and porcelain) and interface 2 (the contact surface which is near to porcelain between zirconia and porcelain) of discs are lower than those at the disc surface (top and bottom). Fig. 2 shows the mean value and standard deviation of biaxial stress at  $r = 0$  (center of disc). The biaxial stress through the disc thickness is shown in Fig. 3. The positive and negative values represent the tensile and compressive strength, respectively. There are two stress zones, the compressive force zone ( $0 \sim 0.6$  mm thickness) and the tensile force zone ( $0.6 \sim 1.6$  mm thickness). Because of the different elastic properties between the two layers, the stress is discontinuous at the interface and the stress gradients are different between the two layers.

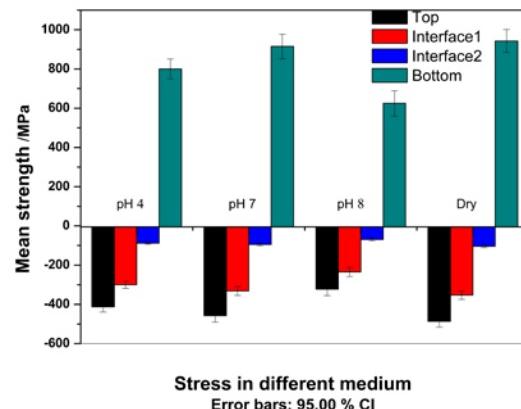


FIGURE 2 MEAN VALUES AND STANDARD DEVIATIONS OF STRESS (MPA) AT FAILURE OBTAINED FOR DIFFERENT INTERFACES OF CERAMIC DISCS WHEN PORCELAIN IS IN THE TOP LAYER

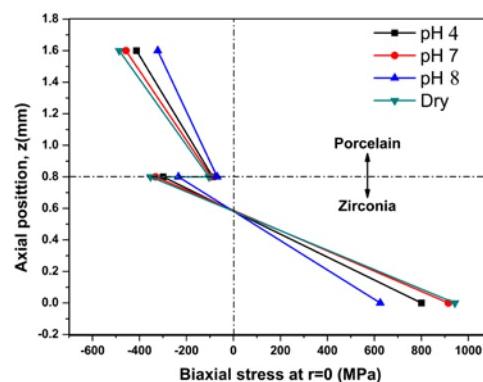


FIGURE 3 BIAXIAL STRESSES OBTAINED FOR DISCS IN DIFFERENT MEDIA. THE BIAXIAL STRESS THROUGH FOR THE PORCELAIN/ZIRCONIA BILAYERED OF A DISC IS OBTAINED UNDER PISTON-ON-RING LOADING WITH PORCELAIN IN THE TOP LAYER

### Damage Maps Analysis

Damage maps analysis is a characterization tool to detect the types of crack formed by cyclic loading.

Subcritical crack growth is notable for its extreme sensitivity to applied stresses (Esquivel-Upshaw et al, 2001). Fig. 4 shows the influence of cyclic loading on crack modes of bilayered structures in both wet and dry conditions. Fig. 4a shows contact damage on the surface after 10,000 cycles in dry air. Fig. 4b shows that radial cracks (RC) are generated outside Hertzian cone cracks; circumferential ring cracking (CRC) is formed outside the indentation and the subsequent secondary radial cracking (SRC) is formed outside the CRC after 10,000 cycles in pH 4. In Fig. 4c the sub-critical radial cracks extend to the edge of the disc after 10,000 cycles in pH 8. Fig. 4d shows contact damage on the surface after 10,000 cycles in pH 7. Fig. 4e displays a cross-section of the Hertzian cone cracks, which are under the contact region of the indenter. The Hertzian cone crack as observed in Fig 4e expands downward as a relatively high rate during fatigue testing or biaxial strength testing. The angle of the outer cone crack relative to the occlusal surface is about  $22 \pm 5^\circ$  and that of the inner cone relative to the occlusal surface is about  $55 \pm 15^\circ$ , which expands downward at a relatively high rate and a sharp angle. Note that secondary radial cracking starts from the top circumferential ring cracking. It is different from the bottom-initiated radial cracking in that it does not pass through the indentation (Wang and Darvell, 2007).

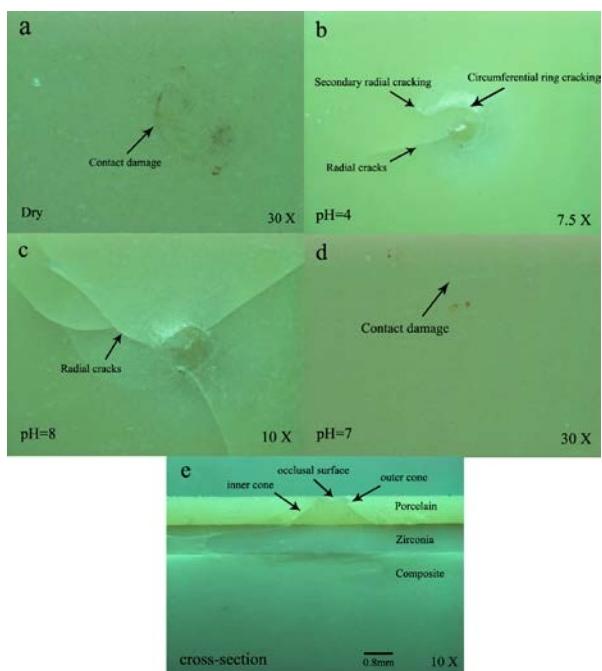


FIGURE 4 OPTICAL MICROGRAPHS ILLUSTRATING TOP (OCCLUSAL) AND SIDE (SECTION) VIEWS OF SURFACE DAMAGE INDWELLING IN BILAYERED DISCS CEMENT-BONDED TO DENTAL COMPOSITE SUPPORT FOLLOWING P = 600 N AND 10000 CYCLES LOADING WITH A SPHERE INDENTER (R = 3.18 MM)

### Weibull Analysis

Most ceramics are reported to have values of the Weibull modulus in the range of 5 ~ 15 (Johnson, 1981). In this study, the Weibull modulus of specimens is averagely 17.75, 17.88, 10.53 and 17.92 in pH 4, pH 7, pH 8 and dry environment respectively (Table 2). A higher Weibull modulus reflects smaller or fewer defects, thus greater structural reliability (Ritter, 1995).

TABLE 2 THE WEIBULL MODULUS OF THE DIFFERENT PART OF THE SPECIMENS

	pH4	pH7	pH8	Dry
Top	17.735	17.87	10.533	17.921
Interface 1	17.737	17.855	10.536	17.907
Interface 2	17.789	17.901	10.522	17.929
Bottom	17.74	17.903	10.534	17.921
Average	17.75	17.882	10.531	17.92

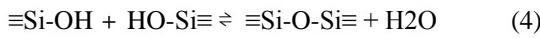
### Discussion

It is found from the experimental results that the flexural strength of specimens tested under the dry condition is approximately 7 ~ 50% higher than that of specimens tested in a wet environment; the flexural strength of specimens under dry condition is approximately 7% higher than that of specimens in neutral solutions (pH=7); the flexural strength of specimens in acid solutions (pH=4) is approximately 30% higher than that of specimens in alkaline solutions (pH=8). These findings show that the changes in flexural strength under cyclic loading are closely associated with the humidity and pH value.

Firstly, the flexural strength of the material is impaired by water. It is known that none of the examined porcelains is chemically inert, even in a neutral aqueous environment. The causes may be chemical and physical actions which result in slow crack growth. On one hand, the reaction between water molecules and Si-O-Si bonds results in chemical destruction, which is one of the mechanisms for slow crack propagation. As explained in (Suputtamongkol, 2003), the process is described as follows: (1) formation of a hydrogen bond between a water molecule and Si-O-Si bonds at the crack tip; (2) interaction of the lone pair of electrons from the oxygen atom in water ( $O_w$ ) and the Si atom; (3) formation of two new bonds between  $O_w$  and Si, and between the hydrogen and oxygen atoms from the silica molecule; (4) rupture of the bonds between hydrogen and  $O_w$  leads to Si-O-H groups on fracture surfaces. These Si-O-H groups will not reform if there is no external energy being supplied.

Secondly, flexural strength degradation may be related to the dissolution of porcelain surfaces promoted by an alkaline or acidic storage medium. The composition of ceramic includes various oxides that can be divided into three categories (alkaline oxide, acidic oxide, neutral oxide). The contents of the various components in the bilayered ceramic discs of our experiment are as follows: SiO<sub>2</sub> 60.0 ~ 70.0%; Al<sub>2</sub>O<sub>3</sub> 7.5 ~ 12.5%; K<sub>2</sub>O 7.5 ~ 12.5%; Na<sub>2</sub>O 7.5 ~ 12.5%.

When the discs are soaked in acidic or alkaline solution, there will be some chemical reactions which will result in the decomposed erosion of the discs. The chemical reaction processes are as follows.



The chemical reaction equations 1, 2, 3 and 4 are the major chemical reaction processes of the discs in acidic solution. The chemical reaction equation 5 is the major chemical reaction processes of the discs in alkaline solution. According to the chemical reaction equations 1, 2 and 3, the surface of the discs will form  $\equiv\text{SiOH}$  layers in acidic solution. The insoluble protecting films  $\equiv\text{Si-O-Si}\equiv$  subsequently is formed through polymerization reaction of  $\equiv\text{SiOH}$  in acidic solution (eq. 4). The insoluble protecting films  $\equiv\text{Si-O-Si}\equiv$  can prevent the decomposed erosion by acidic solution. However, both the rupture of  $\equiv\text{Si-O-Si}\equiv$  and the increase of  $\equiv\text{SiO}^-$  result in the structural damage and continual dissolution of SiO<sub>2</sub> of the discs in alkaline solution (eq. 5). And the insoluble protecting films  $\equiv\text{Si-O-Si}\equiv$  cannot be formed in alkaline solution, too. All of those factors result in the increase of radial cracks in pH 8 (Fig. 3(c)) and finally in a decrease of strength than that in pH 4 (Fig.2 and Fig. 3(b)).

All of these cracks grow slowly in cyclic loading with time, but the inner cone is made larger in pH 8 than that in both pH 4 and pH 7 or under the dry condition by hydraulic pumping and chemical etching. At the same time, the number and length of radial cracks increase with the increase of inner cone cracks. The strength of the disc decreases with the increase in radial cracks.

It should be pointed out that adjustments should be made when using the results of the present study for

predicting clinical behavior of porcelains, because the oral environment presents differences from the storage media used here. In order to obtain authentic data, further study is needed to simulate the real and complex oral environment.

## Conclusion

The main influence of storage medium on bi-axial flexural strength of layered ceramics is investigated in this study. The study found that the flexural strength of specimens tested under dry conditions is approximately 7 ~ 50% stronger than that of specimens soaked in the solution; and the flexural strength of specimens soaked in an acid solution (pH=4) is approximately 30% greater than that of specimens soaked in an alkaline solution (pH=8). The Weibull modulus of specimens is approximately 17.73, 17.88, 10.53 and 17.92 in pH 4, pH 7, pH 8 and dry environment respectively. Thus, the specimens have greater structural reliability in pH 4, pH 7 and dry environment than that in pH 8. The chemical etching of aqueous solution is one of the main influencing factors on the bi-axial flexural strength of layered ceramics in water, especially in alkaline water. So how to avoid chemical etching of aqueous solution is one of the challenges in applications of dental layered ceramics.

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